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NOLTR 65-84

INFRARED ANALYSIS OF THE POLYMERIZATION AND LONG-TERM STABILITY OF EPOXY RESINS

NOL

16 JUNE 1965

UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

NOLTR 65-84

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NOLTR 65-84

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Prepared by:
Doris V. Steele

ABSTRACT: Infrared analysis of epoxy resins utilizes the known fingerprint for these polymers at 2.95, 10.93 and 11.6 microns bands. Five epoxy compounds were studied and the polymerization monitored during 18 months storage time. Three systems reached a stable condition in less than two months, and two systems were still unstable after 18 months storage time. These data correlate extremely well with electrical resistivity data obtained for three of the systems studied. Both electrical resistivity data and infrared analysis provide a sensitive means of determining the long-term stability of epoxy resin systems.

PUBLISHED AUGUST 1965

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WHITE OAK, SILVER SPRING, MARYLAND

NOLTR 65-84 16 June 1965

INFRARED ANALYSIS OF THE POLYMERIZATION AND LONG-TERM STABILITY OF EPOXY RESINS

This report covers a study of the polymerization of epoxy resins by infrared analysis techniques. The results of this study provide an indication of the sensitivity of electrical resistivity data, as compared with infrared data, in determining the completion of the polymerization process and stability of the polymer structure for epoxy resin systems. This work was performed under Task No. NOL-417.

Commercially obtainable materials are discussed throughout this report. Obviously not all candidate materials could be evaluated for the subject program. The selection of a given material for polymerization studies does not constitute an endorsement by the Navy. Neither is this consideration of the material by the Navy to be used for promotional purposes.

R. E. ODENING
Captain, USN
Commander

Albert Lightbody

ALBERT LIGHTBODY
By direction

CONTENTS

ILLUSTRATIONS

REFERENCES

(a) Dannenberg, Hans, and Harp, W. R., Jr., "Determination of Cure and Analysis of Cured Epoxy Resins," Analytical Chemistry, 28, 86 (Jan 1956).

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INTRODUCTION

1. The polymerization of epoxy resins involves complicated chemical reactions which convert the thermoset resin from a soluble liquid into an insoluble solid. This reaction is commonly referred to as the "curing" of the resin. The liquid resin, when completely mixed with a catalyst or hardener, first solidifies in a matter of hours at a relatively slow rate. Then additional crosslinking within the polymer structure takes place at an even slower rate until the polymerization process is completed for all practical purposes and stability of the structure is obtained. In some cases this latter process may require months or even years.

2. Normally, a minimum energy input over a definite time period is required to establish the conditions which will result in a solid resin with stable properties. Decreasing the temperature, for example, reduces this energy input and therefore necessitates an increase in the time of cure. As a result, the polymerization of epoxy resins at room temperature may require extended lengths of time to reach completion. Also, the type and amount of the curing agent used will affect the mechanism as well as the time of the curing process.

3. The degree of cure or the extent of polymerization of epoxy resins can be determined by various methods. For example, the percent conversion of the epoxy groups has been determined by chemical analysis (refs. (a), (b) and (c)). From electrical resistivity data, the activation energy for the electrical conduction process may be calculated and used as an index of the degree of crosslinking within the polymer structure (refs. (d), (e) and (f)). Also, infrared analysis is an established means of monitoring the chemical changes within epoxy resins during cure (refs. (a), (g) and (h)). These latter two methods are nondestructive and, therefore, are easily applied to long-term studies of polymerization versus time. They differ in one important aspect: electrical resistivity data may be obtained from large masses of resin, including filled compounds, whereas infrared data is obtained from a very thin film of the resin only. For many applications, it would be helpful to know the extent of cure in the resin as it is used in a particular unit. This would not be feasible with infrared techniques but could readily be determined by embedding electrodes within the resin mass and obtaining electrical resistivity data at any point in time.

4. The purpose of this study of the polymerization of epoxy resins by infrared analysis was twofold:

a. To determine the mechanism and extent of polymerization of epoxy resins, as indicated by infrared spectra during an extended period of time, to find out if and when the polymerization process was completed and stability of the polymer structure was obtained.

b. To determine the correlation, if any, of the infrared data obtained with the electrical resistivity data already collected and reported in references (e) and (f).

The results of this study provide an indication of the sensitivity of electrical resistivity data, as compared with infrared data, in determining the completion of the polymerization process and stability of the polymer structure for epoxy resin systems.

5. The epoxy resins included in this study were commercially available, inert filled compounds. They were selected as typical systems which exhibit medium, low, and negligible exothermic reactions during the initial polymerization process at room temperature or at low temperature oven curing.

6. The infrared analysis of epoxy resins utilizes the known fingerprint for these polymers at 2.95, 10.93 and 11.6 microns bands. Three epoxy resin systems were shown to be stable after limited room temperature storage or oven curing. Two systems were shown to be still in the process of polymerization after 18 months storage.

7. The electrical resistivity data (refs. (e) and (f)) for three of the five compounds studied by IR indicate the stability or nonstability of the polymer structures to be the same when determined by either infrared or electrical resistivity techniques. Therefore, it may be concluded that both electrical resistivity data and infrared analysis provide a sensitive means of determining the long-term stability of epoxy resin systems.

EXPERIMENTAL WORK

8. Materials. The epoxy resins included in this study were inert filled compounds available commercially. Basically, each is a bisphenol-epichlorohydrin epoxy resin, cured with an amine type curing agent and containing various inorganic fillers. Their selection was based on the recommendations of reference (i), which indicated that they were suitable for various naval ordnance applications. Three of the selected materials had previously been included in the polymerization studies by electrical resistivity techniques, as reported in references (e) and (f). Mixing procedures and the commercial suppliers for all the included compounds are given in Appendix A.

9. Infrared Instrumentation. The infrared spectra were obtained on a Perkin-Elmer Spectrophotometer Model 21. The Model 21 is a recording double-beam spectrophotometer with an optical-wedge attenuator which serves to equalize the energy when absorption occurs in the investigating beam. This instrument is completely described in references (j) and (k). All samples were scanned through sodium chloride windows between the wavelengths of 2 to 15 microns.

10. Experimental Procedures. The epoxy resin and catalyst (or hardener) were thoroughly mixed by means of an electric stirrer for five minutes. After complete mixing, a small portion of the catalyzed resin was poured into a 3 ml conical centrifuge tube to a depth of one inch. The resin was centrifuged for ten minutes to remove the filler. A drop of the supernatant liquid was assembled between two new sodium chloride windows, without spacers, and an infrared spectrum obtained immediately. Time for the experiments was calculated from the first moment that the resin and catalyst were mixed.

11. Additional infrared spectra were obtained for each sample at various intervals of time, utilizing the original assembled sample, allowing the resin to polymerize between the windows. The samples were stored in a desiccator at room temperature between each IR scanning. In the case of oven cured samples, the assembled unit was placed in a heated desiccator (Precision Scientific Co. Heated Vacuum Desiccator, Catalogue No. 68351) at atmospheric pressure and removed from the desiccator for the IR scanning only. Subsequent to oven curing, samples were stored in the desiccator with the other samples at room temperature.

EXPERIMENTAL TEST RESULTS

12. The infrared spectra for the five materials studied are given in Figures 1 through 5. These figures give the complete scanning between the wavelengths of 2 to 15 microns. The initial spectra are shown by the heavy lines. Additional spectra were obtained during 18 months storage of the sample cells to determine if and when stability of the polymer was obtained. The thin lines on Figures 1, 2 and 5 are the spectra showing the disappearance of the epoxy bands and indicating that polymerization was complete for these materials. The thin lines on Figures 3 and 4 show the presence of epoxy bands after 18 months polymerization, indicating a lack of complete polymerization.

13. The progressive polymerization of each material during storage time is shown by Figures 6 through 10. Polymerization was monitored primarily by the disappearance of the epoxy bands at 10.93 and 11.6 microns, and by the appearance of the hydroxyl groups at 2.95 microns. For this reason, Figures 6 through 10 show only that part of the total spectra obtained which covers these ranges of microns, showing the progressive changes with time. Figure 7 shows only the changes at 2.95 microns range as the transmission at the epoxy bands was too low for proper analysis of absorption changes.

14. Hapex 1225 Maskast was almost completely cured after one month as shown by Figure 6. Little change was noted during the next 18 months storage.

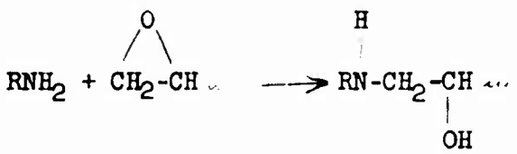
15. Figure 7 data indicates Castaplast 246 to be nearly stable after the first 24 hours polymerization.

16. Figures 8 and 9 show the presence of the epoxy bands in both Stycast 2651 and Stycast 2651MM after 18 months room temperature storage of these compounds, indicating that these materials are still not stable.

17. Oven curing of Scotchcast 502 for 18 hours at 66°C produces a fairly stable polymer with very little change taking place during 18 months storage time, as shown by Figure 10.

DISCUSSION

18. Infrared Analysis Data. The infrared analysis of epoxy resins utilizes the known fingerprint for these polymers. The absorption bands attributed to the epoxy ring linkage are 10.93 and 11.6 microns. The reaction between primary amines and epoxy groups is:



As the polymer cures, the epoxy ring is opened. As a result, the epoxy absorption bands disappear and the appearance of the hydroxyl band at 2.95 microns may be noted. Therefore, these three bands, 2.95, 10.93 and 11.6 microns, may readily be used to determine the rate of polymerization of the polymer system.

19. Examination of Figures 6 through 10 will show the rate at which polymerization has occurred. Stability was noted when the epoxy bands had disappeared and the hydroxyl bands became reproducible.

20. Electrical Resistivity Data. The data obtained from this infrared analysis study may be compared with the electrical resistivity data reported in references (e) and (f) as follows:

a. Hapex 1225 Maskast was considered stable after two months polymerization at room temperature by references (e) and (f). This correlates with the infrared data in that stability was almost reached after one month's storage.

b. Castiplast 247, stable after two months by electrical resistivity data, appears to have reached complete stability in this same time by infrared data shown by Figure 7.

c. Stycast 2651 and 2651MM are similar compounds, differing only in viscosity of the liquid resin. Only Stycast 2651MM was studied by electrical resistivity techniques. Results indicated this polymer to be still in the process of polymerization after sixteen months storage. This data correlates with the infrared data indicating incomplete polymerization at 18 months storage time.

d. Scotchcast 502 was not included in the studies reported in references (e) and (f).

CONCLUSIONS

21. Infrared analysis of epoxy resins may readily be used to monitor the polymerization during long-term storage of the polymer. The disappearance of epoxy bands and the appearance of the hydroxyl band provide the needed fingerprints for this analysis.

22. Infrared analysis data correlates extremely well with electrical resistivity data in determining stability of the polymer structure. Both methods provide an adequate tool for the analytical chemist.

23. Therefore, it may be concluded that both electrical resistivity data and infrared analysis provide a sensitive means of determining the long-term stability of epoxy resin systems.

APPENDIX A

POTTING COMPOUNDS, MIXING PROCEDURES, AND COMMERCIAL SUPPLIERS OF EPOXY RESINS

A-1. The following compounds were used:

Castiplast 247 Epoxy Resin, with 6.25 PHR Hardener 1[a]
Supplier: National Engineering Products, Inc.
Washington Building
Washington 5, D. C.

Hapex 1225 Maskast Epoxy Resin with 15 PHR Hapex 1226 Tufset
Supplier: Hastings Plastics, Inc.
1551 - 12th Street
Santa Monica, California

Stycast 2651 Epoxy Resin with 8 PHR Catalyst 11
Stycast 2651MM Epoxy Resin, with 8 PHR Catalyst 11
Supplier: Emerson and Cuming, Inc.
869 Washington Street
Canton, Massachusetts

Scotchcast 502 Epoxy Resin, 95 pts by wt Part A with 5 pts by wt Part B
Supplier: Electrical Products Division
Minnesota Mining and Manufacturing Co.
St. Paul 6, Minnesota

NOTE: (a) This material is currently available as:

Casting Compound No. 47, with 6.25 PHR Hardener A
Supplier: Rector Engineering Plastics Co.
318 Randolph Place, N. E.
Washington, D. C.

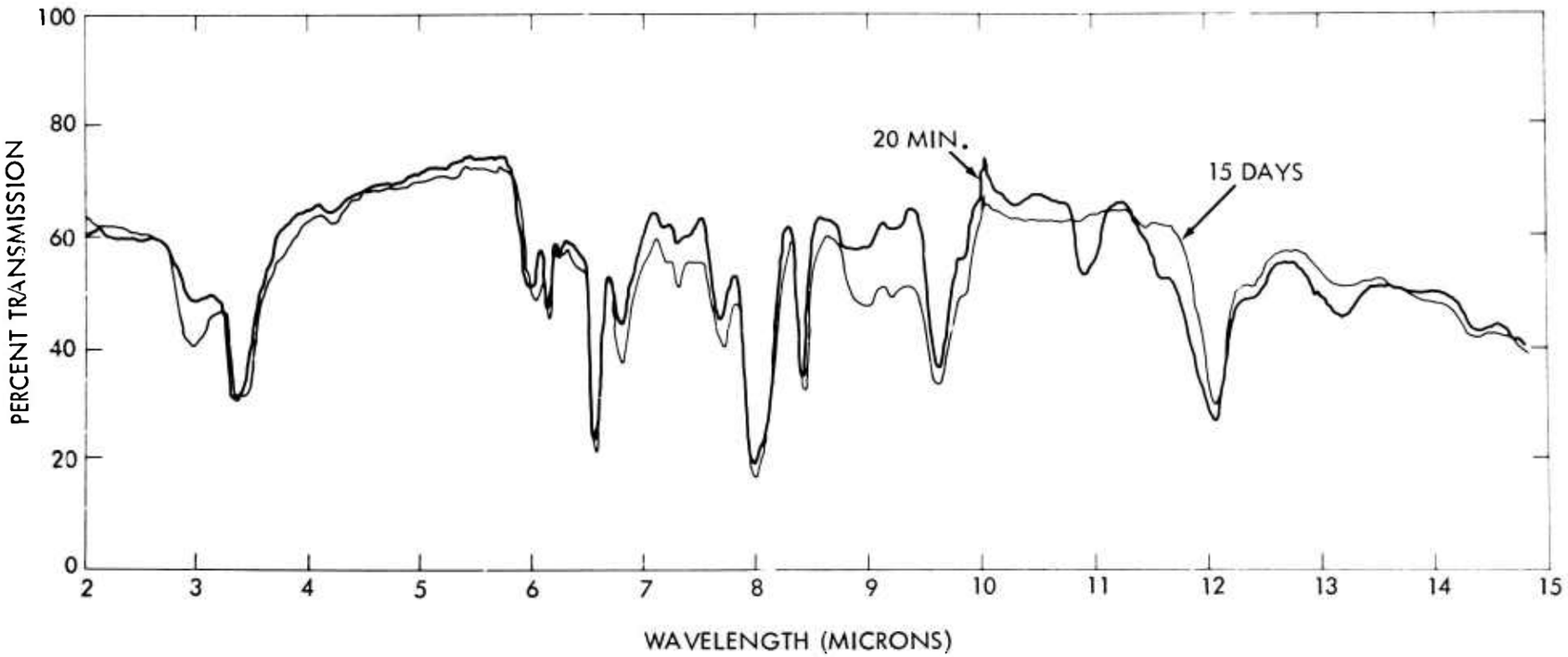


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FIG. 1 INFRARED SPECTRA OF HAPEX 1225 MASKAST, 20 MINUTES AFTER MIXING AND AFTER 15 DAYS AT ROOM TEMPERATURE

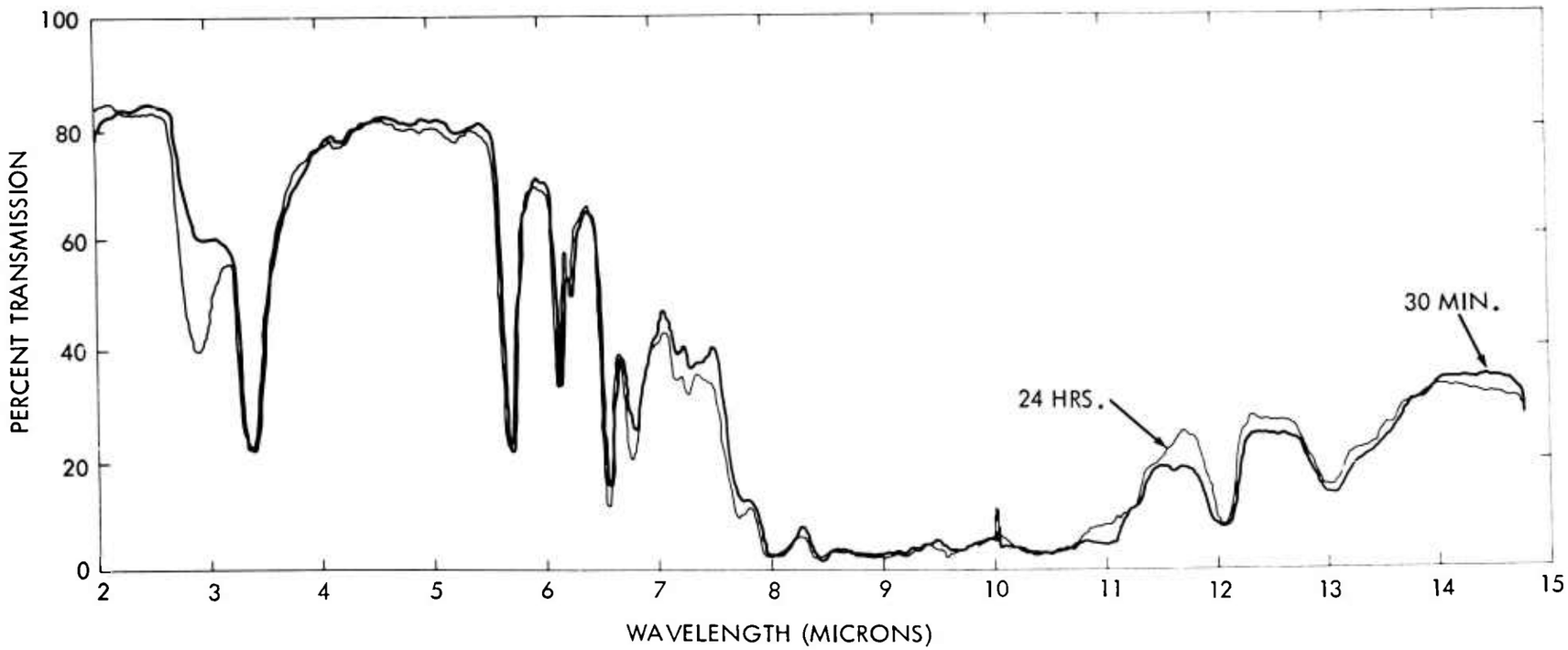

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FIG. 2 INFRARED SPECTRA OF CASTIPLAST 247, 30 MINUTES AFTER MIXING AND AFTER 24 HOURS AT ROOM TEMPERATURE

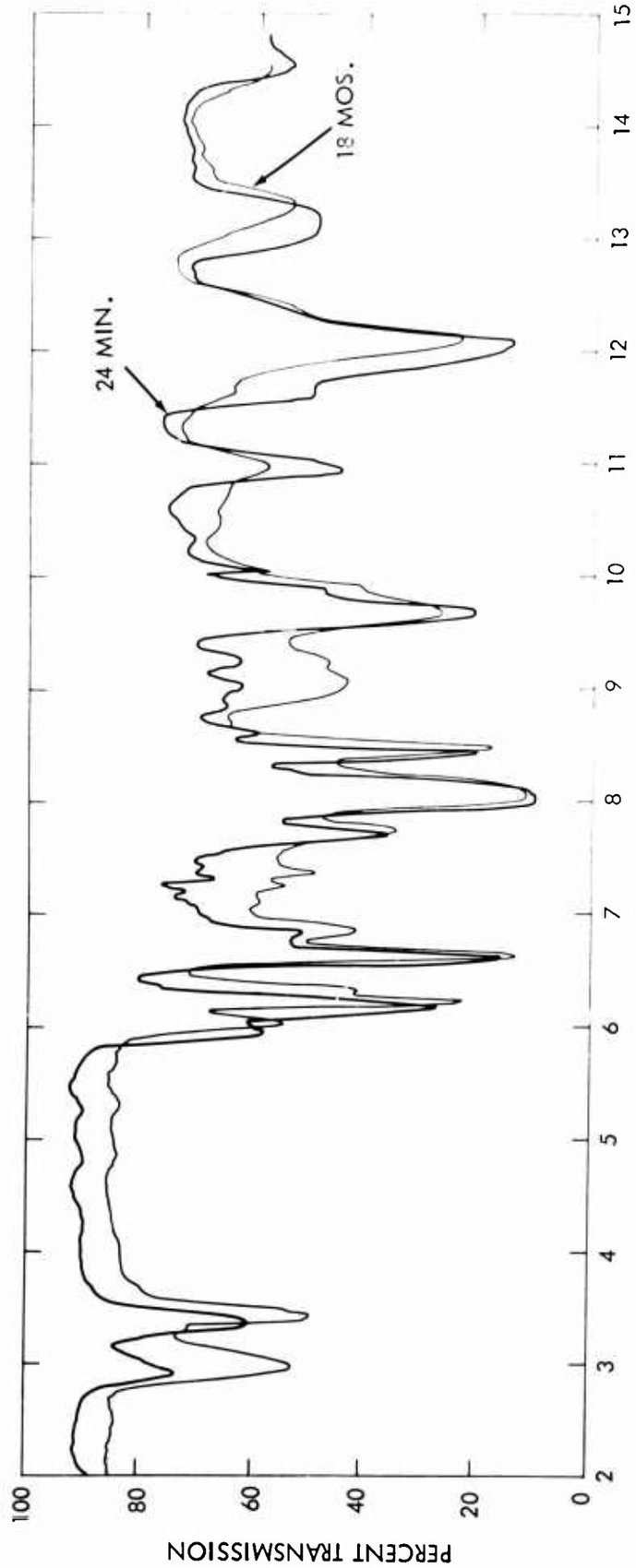

FIG. 3 INFRARED SPECTRA OF STYCAST 2651 WITH CATALYST 11, 24 MINUTES AFTER MIXING AND AFTER 18 MONTHS AT ROOM TEMPERATURE

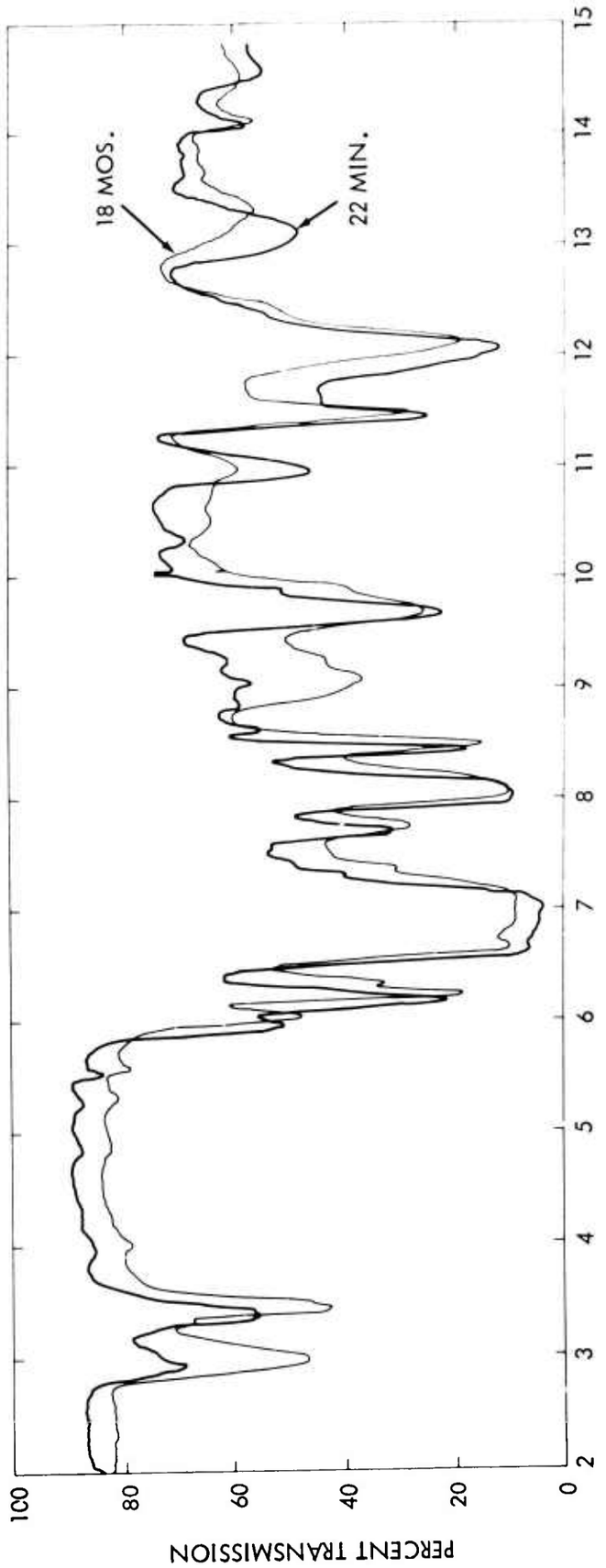

FIG. 4 INFRARED SPECTRA OF STYCAST 2651 MM WITH CATALYST 11, 22 MINUTES AFTER MIXING AND AFTER 18 MONTHS AT ROOM TEMPERATURE

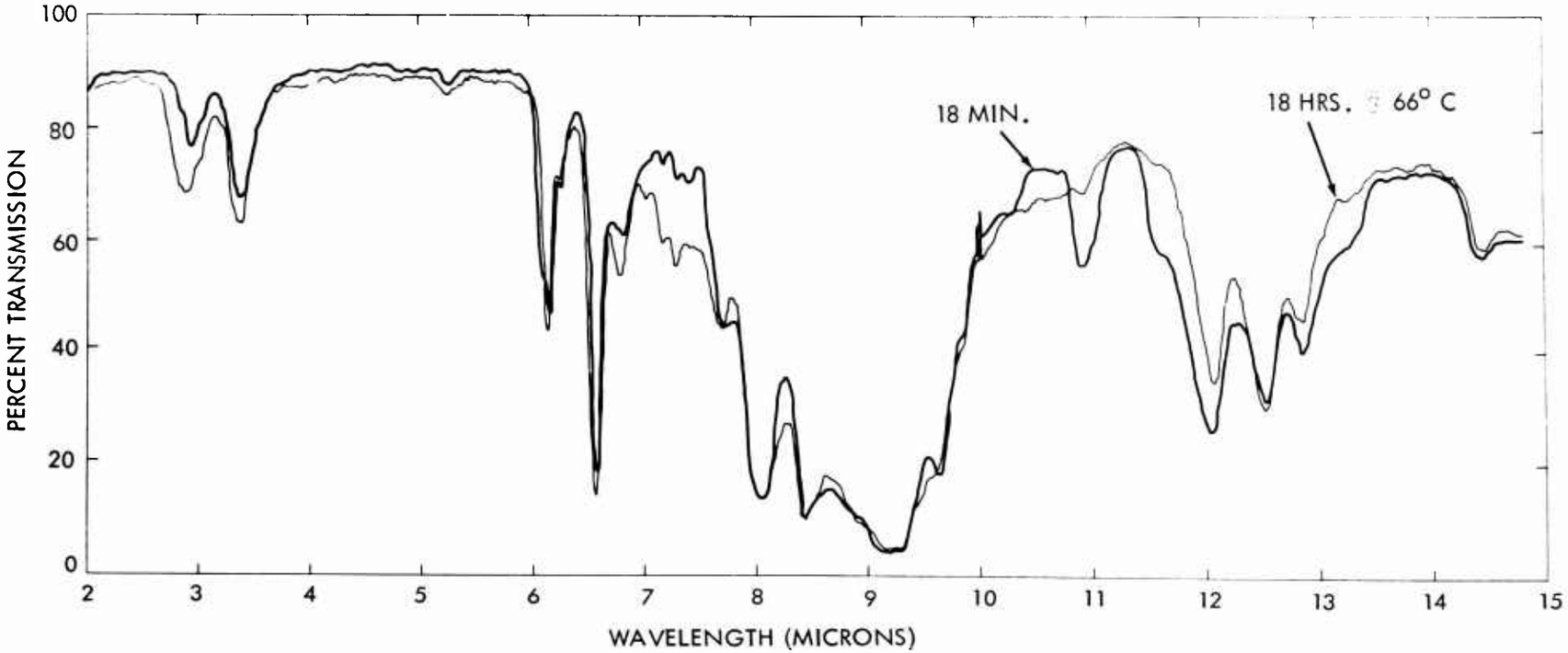

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FIG. 5 INFRARED SPECTRA OF SCOTCHCAST 502, 18 MINUTES AFTER MIXING AND AFTER 18 HOURS OVEN CURING AT 66° C

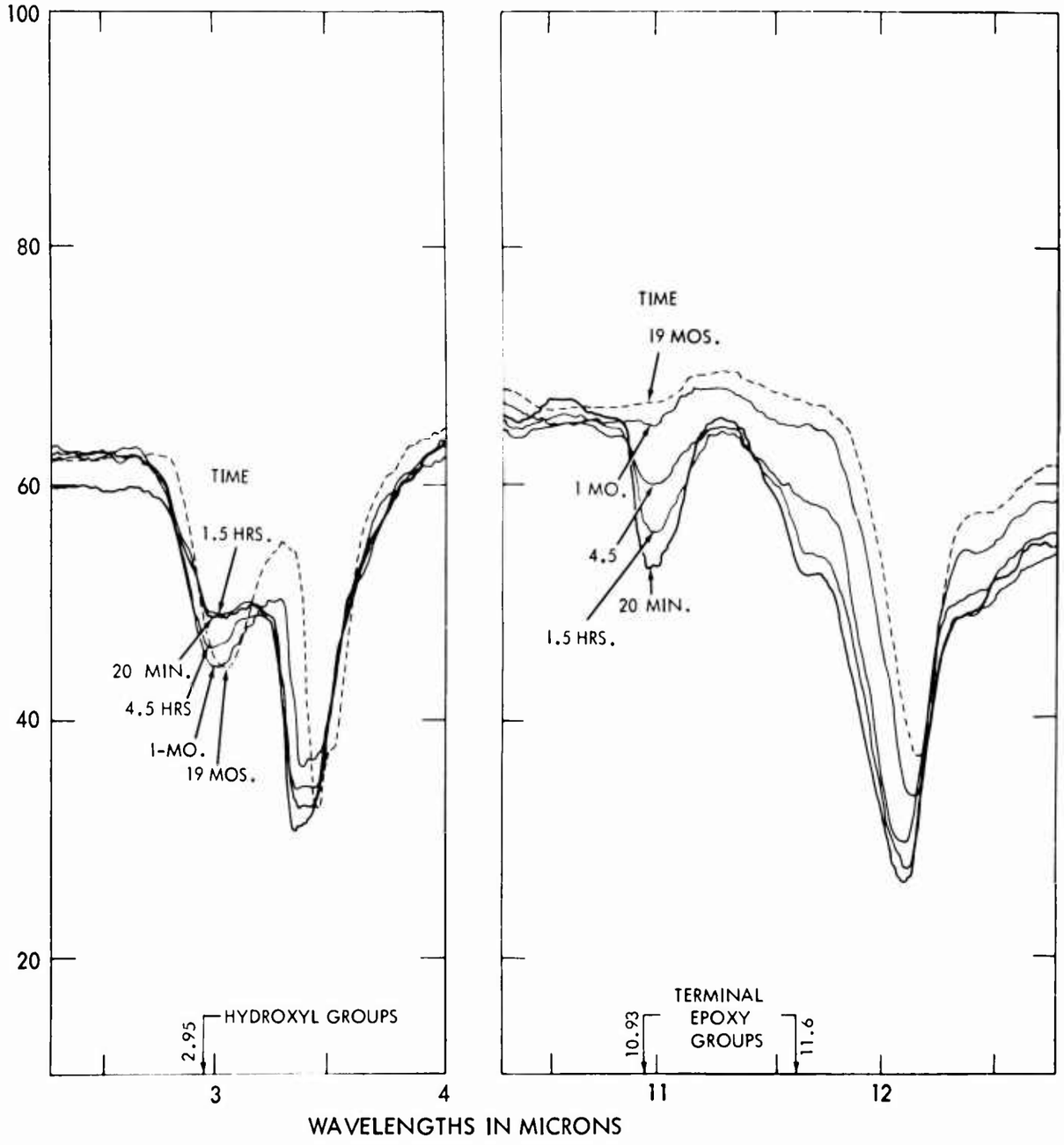

FIG. 6 SPECTRA DURING POLYMERIZATION OF HAPEX 1225 MASKAST, CURED AND STORED AT ROOM TEMPERATURE

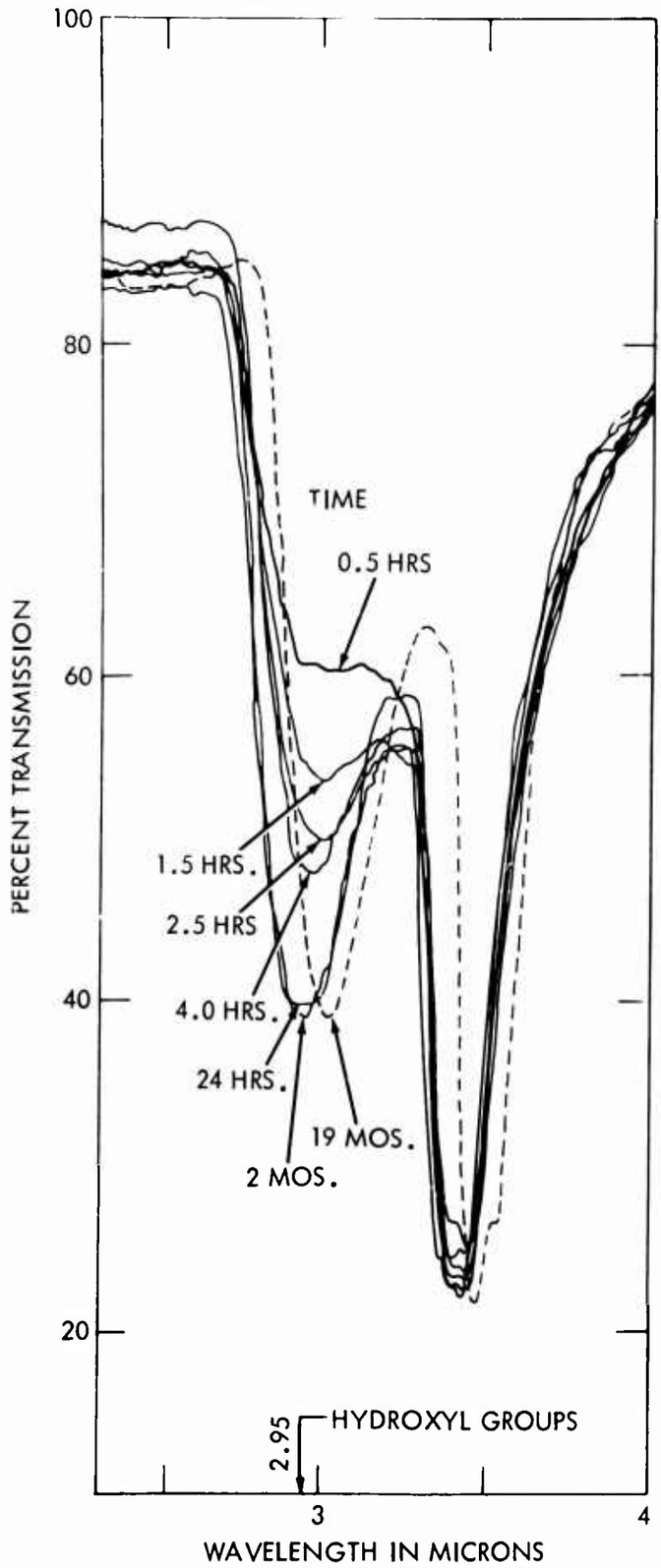

FIG. 7 SPECTRA DURING POLYMERIZATION OF CASTAPLAST 247, CURED AND STORED AT ROOM TEMPERATURE

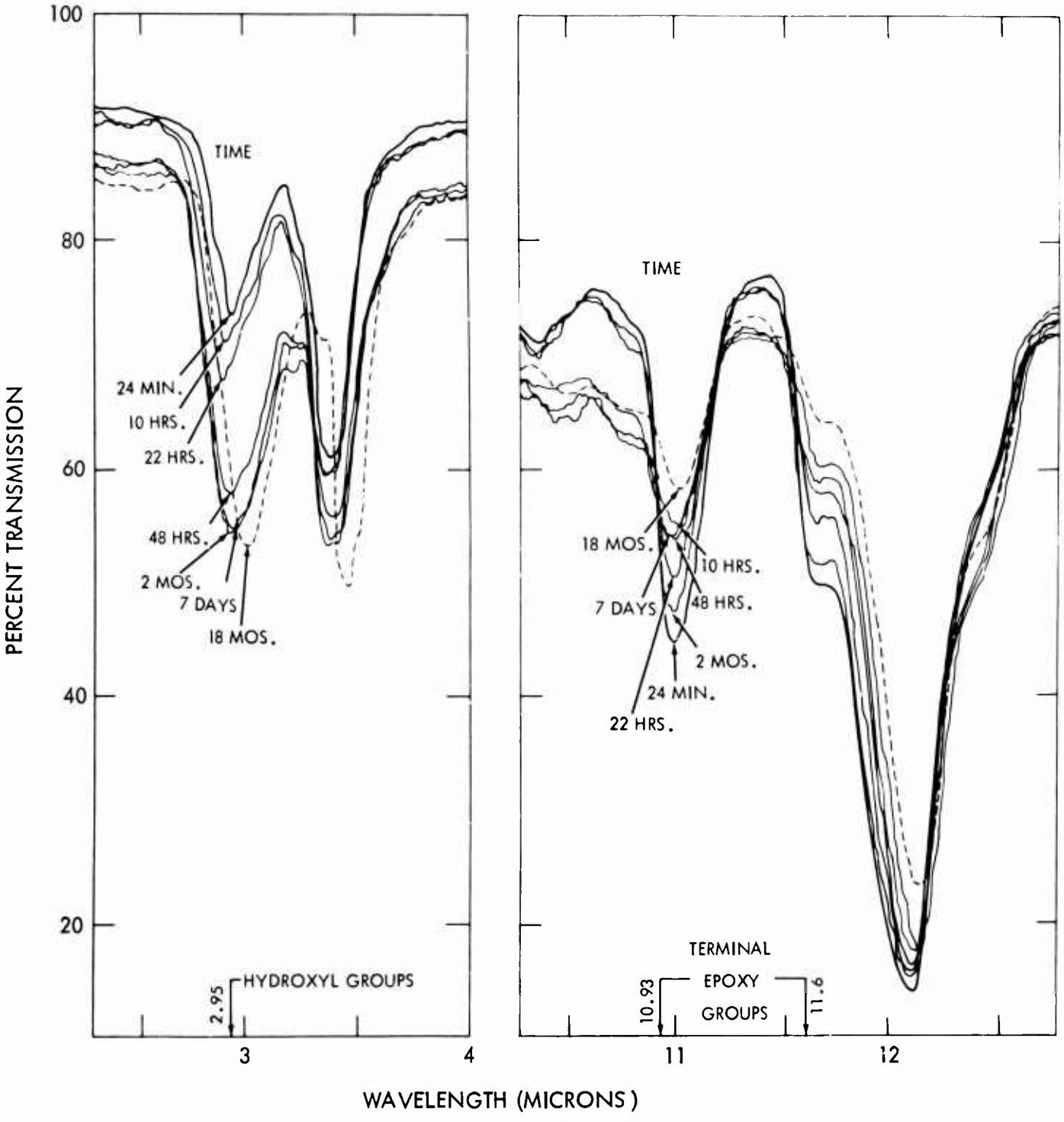

FIG. 8 SPECTRA DURING POLYMERIZATION OF STYCAST 2651 WITH CATALYST 11, CURED AND STORED AT ROOM TEMPERATURE

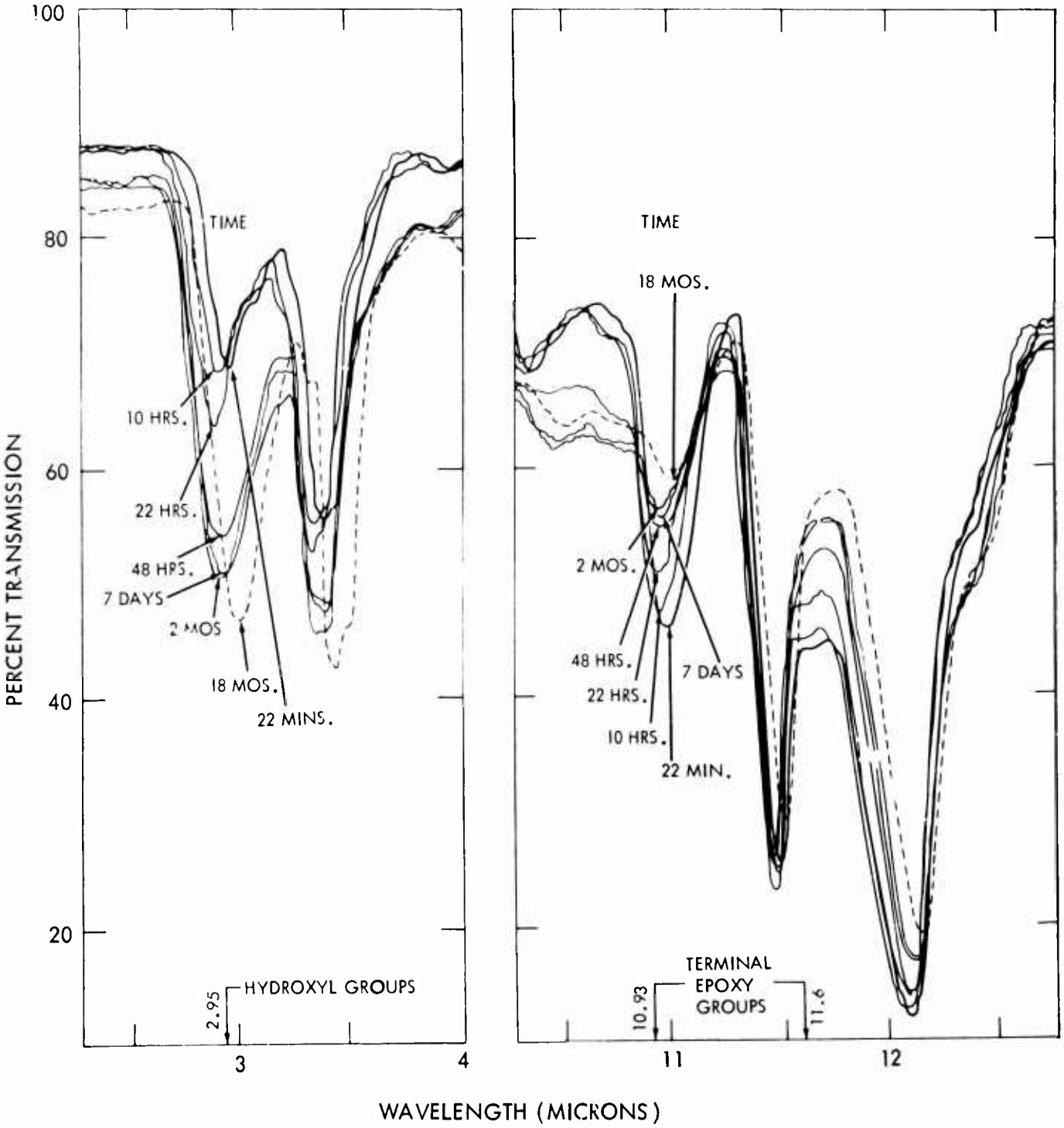

FIG. 9 SPECTRA DURING POLYMERIZATION OF STYCAST 2651 MM WITH CATALYST 11. CURED AND STORED AT ROOM TEMPERATURE

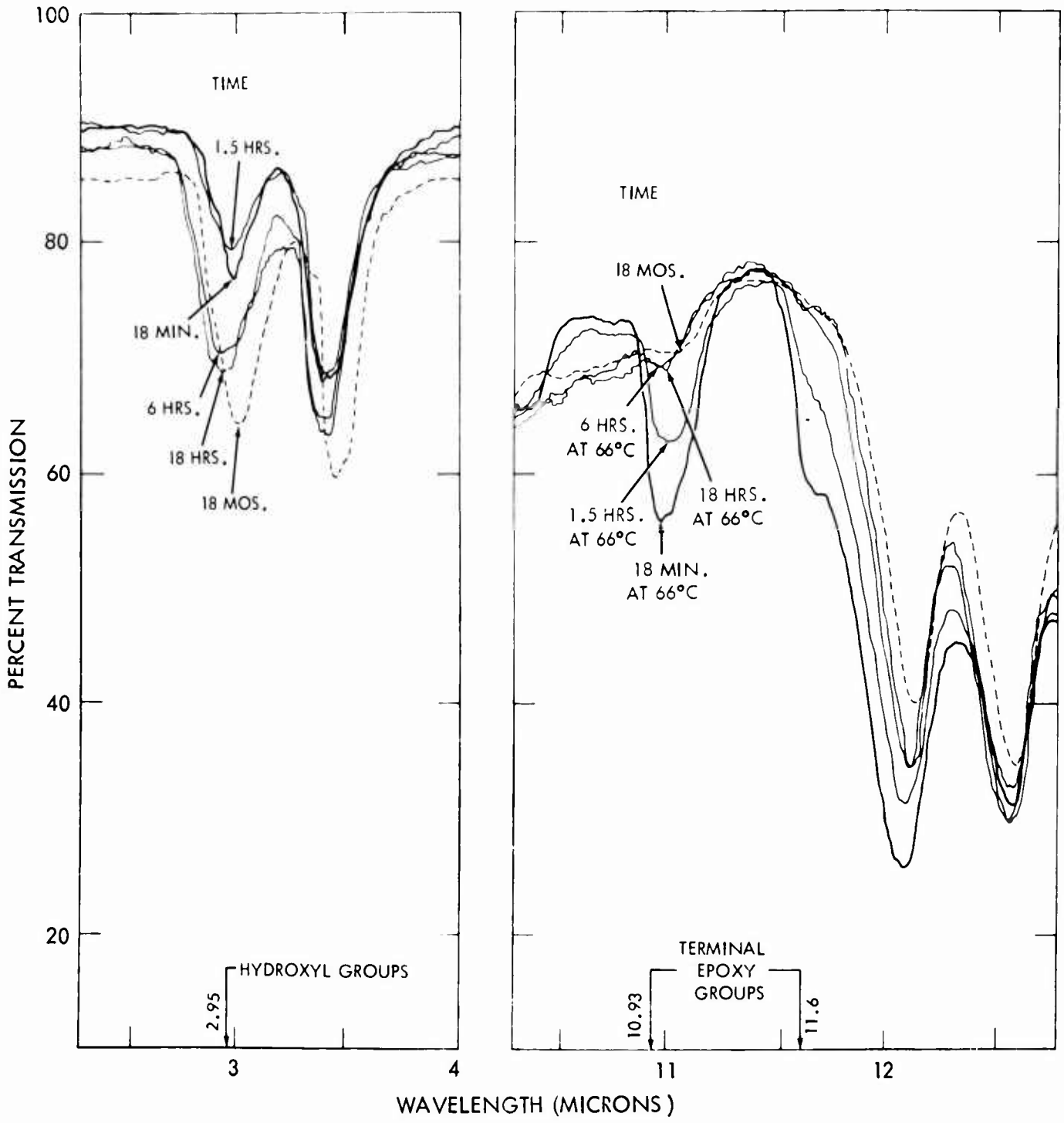

FIG. 10 SPECTRA DURING POLYMERIZATION OF SCOTCHCAST 502, CURED 18 HOURS AT 66° C THEN STORED AT ROOM TEMPERATURE

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(NOL technical report 65-84)
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